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Near-Infrared Spectroscopy for Rapid Determination of Physicochemical Properties in Fermented Fish Sauce ('Nam Pla-Ra')

Running head: Near-Infrared Spectroscopy for Fermented Fish Sauce Determination

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SUMMARY

Research background. Thai community product quality standards have been established for nam pla-ra, which require at least 4 % protein and 12 % salt (sodium chloride). Determining the protein and sodium chloride (NaCl) contents requires the use of chemicals and is time-consuming. In addition to reporting protein and salt content, there are other quality controls that need to be carried out. Thus, the objective of this research was to develop predictive models for the determination of nam pla-ra physicochemical values by using near-infrared spectroscopy (NIRS) for fast and simultaneous multi-parameter prediction.

Experimental approach. The physicochemical values of commercially available fermented fish sauce ('nam pla-ra' in Thai) were evaluated to develop predictive models for physicochemical values determination and to develop models to categorize samples. The model was developed and tested based on the seven parameters: total soluble solids (TSS), pH, *L**, *a**, *b**, NaCl, and protein content.

Results and conclusions. The predictive models for pH and color (*L**, *a**, *b**) could be used for

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screening, based on their coefficients of determination (R^2=0.73–0.81; SEP=0.15–0.94); the protein content model could be used for research and general work (R^2=0.86; SEP=0.44); and the TSS and NaCl models could be used for all types of work (R^2=0.97–0.98; SEP=0.41–0.61). Nam pla-ra physicochemical values could be predicted accurately using TSS, color (*L**, *a**, *b**), NaCl, and protein content. There was no significant (p>0.05) difference between the quality value from the predictive model and the reference method, except for the pH value.

Novelty and scientific contribution. NIRS technique showed a potential for nam pla-ra physicochemical values prediction using TSS, color (*L**, *a**, *b**) with the whole range from 1000–2500 nm, and NaCl and protein contents with the selected range from 1000–1889.64 and 2030.87–2408.48 nm. Furthermore, these findings demonstrate the potential of NIRS as a rapid, non-destructive, environmentally friendly, and reliable analytical tool for nam pla-ra. NIRS can contribute to ensuring product consistency and facilitating standardization in the fermented fish sauce industry. Such applications enhance food safety and consumer confidence.

Keywords: fermented fish sauce (nam pla-ra); near infrared spectroscopy; physicochemical values determination; predictive model

INTRODUCTION

Fermented fish ('pla-ra' in Thai) is produced from freshwater fish, such as *Channa striata* ('chon' in Thai), *Trichogaster trichopterus* ('kra-dee'), *Cyclocheilichthys repasson* ('soi'), and *Puntius gonionotus* ('tapien'), which are fermented with salt and rice bran or roasted rice powder in a closed container for 6 to 12 months at ambient temperature. This method is important for the preservation of high protein foods (*1,2*).

A product called fermented fish sauce ('nam pla-ra' in Thai) is made from pla-ra and is seasoned with ingredients including sugar, tamarind juice, and pickled garlic juice. Then, it is filtered, cooked, and packaged (*3*). In Thailand, particularly in the north and northeast, nam pla-ra is primarily used as an ingredient in regional dishes, such as curries, soups, and spicy salads.

Initially, small- and medium-scale manufacturing of pla-ra and nam pla-ra focused on domestic or local consumption. Pla-ra and nam pla-ra are in great demand due to migration and the sharing of food cultures. Pla-ra production in Thailand is approximately 40,000 tonnes per year, worth almost THB 800 million per year, while the value of pla-ra for export (to Laos, Vietnam, Cambodia, the United States, the European Union, and Middle Eastern nations) is more than THB 20 million per year (*4*). Consequently, Thai community product quality standards have been established for nam pla-ra, requiring it to include at least 4 % protein and 12 % salt (sodium chloride) (*3*).

Determining the protein and sodium chloride (NaCl) contents requires the use of chemicals and is time-consuming; consequently, near-infrared spectroscopy (NIRS) is a powerful tool for determining physicochemical values that is rapid, practical, environmentally safe, and requires little sample preparation. This technique can be used for both quantitative and qualitative studies of food products and it can be used to determine several physicochemical values in a single scan (*5–8*). Other studies have successfully evaluated the quality of alcoholic and acid fermentations from low-grade fruits using NIRS (*9,10*). However, other fermented products having nontransparent and thick characteristics, such as nam pla-ra sauce are very challenging to investigate using the NIRS method. In fish or other sauces, NIRS can be used for the physicochemical values determination of parameters, such as total soluble solid (TSS), total nitrogen content, and pH of fish sauce (*11*), final quality of soy sauce (shoyu) (*12*), total nitrogen content in soy sauce (*13*), and sensory quality of Japanese fermented soy bean paste (miso) (*14*). However, no published studies have mentioned the use of NIRS models for predicting nam pla-ra physicochemical values, such as the protein and NaCl contents, which are important parameters to be controlled based on standards. In addition to standard efforts, further quality control parameters of TSS, pH, and color (L^*, a^*, b^*) were also included in this study. Thus, the objective of this research was to develop predictive models of nam pla-ra physicochemical values by using NIRS for fast and simultaneous multi-parameter prediction.

MATERIALS AND METHODS

Material

Three brands of nam pla-ra with three degrees of sodium chloride concentration and protein content (low, medium, and high) were chosen from regional and local supermarkets in Bangkok and Pathum Thani Province, Thailand. They were used without further preparation and with a mixing ratio by the mixture design technique for a total of 60 samples. Additional forty brands from the markets were acquired. All samples (N=100) were used to create the calibration and prediction models for nam pla-ra physicochemical values. The information on nam pla-ra (manufacturer, city, province, and country of origin) and the details of the mixture design technique are presented in Table S1 and Table S2, respectively.

FT-NIR analysis

Thirty mL of nam pla-ra of each sample were placed in separate plastic tubes and incubated in a water bath at 30 °C for 30 minutes. Then, each sample was placed in a glass Petri dish in combination with a transflection cover and analyzed using an FT-NIR spectrophotometer, model NIR

Flex N-500 (Buchi, Flawil, Switzerland) in the transflectance mode. All spectra were collected in the wavenumber range 10000–4000 cm^{-1} (1000–2500 nm) with the number of sample scans of 64 and resolution of 8 cm^{-1}. A single spectrum was produced for each sample by averaging the three spectra that were acquired.

Physicochemical characterization of nam pla-ra by reference methods

The nam pla-ra physicochemical values—total soluble solid (TSS), pH, color (L^*, a^*, b^*), sodium chloride content (NaCl), and protein content—were determined following reference methods. Three measurements were carried out per sample.

TSS

TSS was measured using a pocket refractometer, model PAL-1 (Atago, Tokyo, Japan).

pH

The pH value was determined at a temperature of 25 °C using a pH meter, model CG842 (Schott, Mainz, Germany).

Color

The color (L^*, a^*, b^*) of each sample was measured using a spectrophotometer, model CM-700d (Konica Minolta, Osaka, Japan).

NaCl

A sample (1 g) was weighed into a beaker along with 200 mL of deionized water to determine the NaCl of the sample. The mixture was adjusted to neutral pH with 0.1M sodium hydrogen carbonate (Elago Enterprises Pty Ltd, New South Wales, Australia) solution and the volume was adjusted to 250 mL with deionized water. A sample (10 mL) was pipetted into an Erlenmeyer flask and 1 mL of 5 % potassium chromate (Elago Enterprises Pty Ltd, New South Wales, Australia) solution was added. The sample was titrated with 0.01M silver nitrate (Avantor Performance Materials Poland Spolka Akcyjna, Gliwice, Poland) solution until the end point (brick red sediment). Finally, the NaCl of the sample was calculated using Eq. 1 (*15*):

$$\text{NaCl (\%)} = [(V1\text{-}V2)\cdot c\cdot 0.05844\cdot 100\cdot \text{dilution factor}]/10 \qquad /1/$$

where *V*1 and *V*2 are the volumes of silver nitrate solution titrated with the sample (mL) and the blank (mL), respectively, and *c* is the concentration of silver nitrate solution (M).

Protein content

The protein content was determined according to the AOAC method (*16*) using the Kjeldahl method. A sample (2 g) was weighed into a protein digestion tube. Subsequently, 10 g of a selenium mixture and 15 mL of sulfuric acid were added. A blank was prepared in parallel using distilled water instead of the sample. The samples were subjected to digestion and then allowed to cool to room temperature. After digestion, the samples were distilled by adding 25 mL of 4 % boric acid solution into a 250 mL flask containing 2 drops of indicator solution, which was used to collect the distillate. The distillate was then titrated with 0.1N hydrochloric acid solution until the endpoint was reached. The volume of 0.1N hydrochloric acid solution consumed was recorded. The protein content was calculated using Eqs. 2 and 3:

$$\text{Total nitrogen (\%)} = [(V1-V2)\cdot c\cdot 1.4007]/m \qquad /2/$$

$$\text{Protein content (\%)} = \text{Total nitrogen (\%)}\cdot 6.25 \qquad /3/$$

where *V*1 and *V*2 are the volumes of hydrochloric solution used in the titration of the sample (mL) and the blank (mL), respectively, *c* is the concentration of hydrochloric solution (N), and *m* is the sample mass (g).

NIR spectroscopic model development

Predictive model development

In the development of predictive models, the standard sequence involves calibration, validation, and prediction. For the purposes of this pilot study, however, the validation procedure was implemented in place of the prediction stage. The measurement data and NIR spectra (full and selected spectra) were separated into two groups consisting of the calibration set (*N*=75) and the prediction set (*N*=25). The Unscrambler software v. 9.8 (CAMO AS, Trondheim, Norway) (*17*) was used to create chemometric models using partial least square (PLS) regression with both the original and pre-treated spectra. Spectral pre-processing included second derivatives (SD) based on the Savitzky–Golay method (polynomial order=2) and standard normal variate (SNV) to reduce signal variation caused by light scattering effects in the samples and to enhance the predictive accuracy and stability of the model. The coefficient of determination (R^2), standard error of calibration (SEC), standard error of prediction (SEP), and bias were used to describe the performance of each model. The optimal model was selected based on lowest SEC, SEP, and bias and the highest R^2.

Predictive model validation

The predictive sample set (*N*=25) was used for validating the predictive model in the Unscrambler software (*17*). After that, statistical tests were done using a paired sample t-test where significance was tested at the $p<0.05$ level between the measured value from the reference method and the predicted model using the IBM SPSS Statistics v. 17.0 software (*18*). The sig. (2 tailed) and bias were used to describe the performance of model.

RESULTS AND DISCUSSION

Physicochemical values of nam pla-ra by reference method

Table 1 displays the characteristics of nam pla-ra as determined by the reference technique in terms of total soluble solid (TSS), pH, color (*L**, *a**, *b**), sodium chloride concentration (NaCl), and protein content. The physicochemical (TSS, pH, color, NaCl, and protein content) and the spectra results were divided into two groups to create the predictive model, with a calibration set (*N*=75) and a prediction set (*N*=25). The ranges of all physicochemical reference values in the calibration set fully encompassed those observed in the prediction set.

FT-NIR analysis

The NIR spectra of nam pla-ra samples were determined using an FT-NIR spectrophotometer in the transflectance mode and the spectral profiles of the samples are plotted in Fig. 1a.

Fig. 1b shows the NIR spectra of samples recorded on FT-NIR in the highest and lowest protein content with the second derivative Savitzky-Golay method pretreatment for important band assignment, while Table 2 shows the band assignment data. Water is a key element in nam pla-ra. At 1403, 1463, and 1877–1900 nm, it exhibited a strong absorption band that is the combination of the O-H overtone, stretching, and bending wavelengths (*19*). The bands associated with pH (lactic acid from the fermented process and the ionization of lactate from lactic acid) were found at 1710, 1722, 1730, 1750, 1890, 2160, and 2470 nm as the first overtones of the C-H stretching, O-H stretch combined with C=O stretch, C-H stretch, C=O stretch combination, and C-H stretch combined with C-O stretching (*20*–*24*). The absorption bands for protein in the spectral region 2050–2168 nm resulted from the N-H stretching combination and the N-H band second overtone (*24*). At 2242 and 2472 nm, there were combinations of C-H stretching, CH2 deformation combination, C-C stretching combination, and C-O-C stretching combination that were related to sugar (*24*). Furthermore, despite NaCl lacking unique NIR absorption bands, the action of the salt on the water absorption band allowed for the detection of changes in salt concentration. The strength of the water band's absorption decreased as the salt content increased (*19,25*). For TSS, it can be attributed to NaCl, as the main soluble solids dissolved in nam pla-ra. NIRS does not directly measure the *L**, *a**, *b** color parameters of fermented fish sauce like human vision or a colorimeter. Instead, it measures light absorption or reflection in the near-infrared range, which is indirectly correlated with the chemical composition and physical structure affecting the color of the product through precise chemometric modeling. In 'nam pla-ra,' the brown to dark brown color is due to compounds containing C-H, O-H, and N-H bonds, which are detectable by NIRS. Variations in these color-forming compounds alter the NIR spectrum. Specifically, their concentrations, which determine *L**, *a**, and *b** color values, correlate with light absorption in the NIR range.

Calibration models of physicochemical values were produced using PLS regression. The calibration and prediction statistics are shown in Table 3, and scatter plots for comparison of the measured and predicted values for each parameter are shown in Fig. 2 (models of the full spectrum in the range of 1000–4000 nm) and Fig. 3 (models of the selected spectrum in the range of 1000–1889.64 and 2030.87–2408.48 nm to avoid overlapping and over-absorption bands (*26*)). The performance of the PLS calibration models was evaluated using the coefficient of determination (R^2), the standard error of calibration (SEC), and the standard error of prediction (SEP). As presented in Table 3, the number of factors represents the optimal number of latent variables used in the PLS regression models, selected to achieve an appropriate balance between model accuracy and complexity while avoiding overfitting. In this study, the optimal number of factors ranged from 4 to 22, depending on the physicochemical parameter and the spectral preprocessing method applied. The use of an appropriate number of factors contributed to minimizing prediction errors, indicating good model stability and generalization capability. Overall, the calibration models exhibited relatively high R^2 values, demonstrating strong agreement between the measured and predicted physicochemical properties. In addition, the SEC and SEP values were generally low and comparable for most parameters, suggesting satisfactory calibration accuracy and reliable prediction performance. For the full spectrum model, the original spectrum produced the best results for pH, color (*L** and *a**), and NaCl. Second derivative of NIR spectra using the Savitzky-Golay method was optimal for color (*b**), while the standard normal variate (SNV) method was optimal for TSS and protein content. For the selected spectrum model, the original spectrum was optimal for TSS, color (*b**), and NaCl. The second derivative of NIR spectra using the Savitzky-Golay method was optimal for pH and protein content, while the standard normal variate (SNV) method was optimal for color (*L** and *a**).

The comparison between the full spectrum and selected spectrum models indicated that the best predictive model from the full spectrum consisted of pH and color (*L** and *a**). In contrast, the best predictive model from the selected spectrum, were developed using TSS, color (*b**), NaCl, and protein content. The whole spectrum provided the best model for physical characteristics, since color may emerge from physical characteristics not having distinct absorption bands in the NIR. The best model for physical parameters was produced using the full spectrum (representative of all functions) predictive model, while the selected spectrum gave the best model for chemical properties (TSS, NaCl, and protein content), perhaps because the chemical properties corresponded to specific absorption bands. Thus, the selected spectrum (without the big band of water) provided the best model for specific absorption bands. Furthermore, the predictive models for pH and color (L^*, a^*, b^*) may be utilized for screening (R^2=0.73–0.81); the protein content models can be used for research and general work (R^2=0.86); and the TSS and NaCl models can be used for all types of work (R^2=0.97–0.98) (*27*).

Compared with previous studies, Ritthiruangdej *et al*. (*11*) applied NIRS in the spectral range of 1100–2500 nm to predict total nitrogen (TN), pH, refractive index, density, and Brix in Thai fish sauces. Their results showed that the informative spectral regions selected by SCMWPLS were strongly associated with specific chemical properties, including the regions of 2264–2428 nm for TN; 1698–1722 and 2222–2258 nm for pH; 1358–1438 nm for density; 1774–1846 and 2078–2114 nm for refractive index; and 1322–1442 and 2000–2076 nm for Brix. In addition, qualitative classification models based on TN content achieved correct classification rates exceeding 82%. These findings are consistent with the results of the present study, demonstrating that variable selection and the use of informative spectral regions can improve the prediction of chemical attributes related to distinct NIR absorption bands, whereas full spectrum models are more suitable for predicting physical characteristics. Similarly, Jiang *et al*. (*28*) investigated the application of dual-band NIRS for the non-destructive determination of fat, protein, collagen, ash, and sodium contents in soy sauce stewed meat. Using spectral ranges of 650–950 nm and 960–1660 nm, PLS models were developed based on spectra collected from vacuum-packed ready-to-eat products from 97 different brands. The results demonstrated that fat and protein contents were predicted with the highest accuracy, with superior model performance obtained in the 960–1660 nm region compared to the 650–950 nm range. These findings are in agreement with the present study, emphasizing that the selection of appropriate spectral regions enhances predictive performance for chemical constituents associated with specific absorption bands. Furthermore, Zhang *et al*. (*29*) collected NIR spectra in the range of 1000–2500 nm using a miniature fiber-optic NIR spectrometer to monitor different stages of soy sauce production, including steamed soybean, koji, and moromi. Their study successfully developed predictive models for in situ and real-time assessment of the digestion rate of steamed soybean, protease activity in koji, and formaldehyde nitrogen content in moromi. These results further support the suitability of NIRS for predicting key chemical attributes in fermented food systems, which is consistent with the findings of the present study. Nevertheless, there have been no reported studies on the quantification of physicochemical properties of nam pla-ra using NIRS.

Predictive model validation

The predictive model validation showed that the created model had accurate nam pla-ra physicochemical values prediction (TSS, color (*L**, *a**, *b**), NaCl, and protein content). There was no significant difference between the physicochemical values from the predictive model and the reference method, except for the pH value (Table 4) perhaps due to the pH value in the sample having a rather narrow range. The pH value (Table 1) had the lowest SD value (0.38) compared to other physicochemical values. Therefore, the accuracy of the pH value might still be impacted by the prediction model, even if it has a low bias value. Consequently, the outputs of the prediction model and the reference method were different.

CONCLUSIONS

In summary, the nam pla-ra physicochemical values consisted of total soluble solid (TSS) (13.93–48.50 %), pH (4.08–6.06), *L** (25.97–36.51), *a** (1.31–7.59), *b** (1.69–10.82), sodium chloride content (NaCl) (8.38–21.87 %), and protein content (1.57–7.82 %). The PLS regressions between the FT-NIR spectra (full and selected) and nam pla-ra physicochemical values were developed. These included TSS, pH, color (*L**, *a**, *b**), NaCl, and protein content. The research findings clearly indicate that for predicting physical properties or parameters involving complex overall composition—such as pH, *L**, or *a** values—the most accurate models are those developed using full-spectrum data. In contrast, for predicting specific chemical parameters such as TSS, NaCl, and protein content, as well as the *b** value (yellow-blue axis), which relates to the chemical components responsible for the characteristic color of fermented fish meat, the optimal models are derived from selected spectral regions. This information will support the immediate application of selecting wavelength ranges that correlate with physicochemical properties. In addition, it was found that pH and color (*L**, *a**, *b**) models could be used for screening, the protein content model could be used for research and general work, and the TSS and NaCl models could be used for all types of work. The created model could accurately predict nam pla-ra physicochemical values (TSS, color (*L**, *a**, *b**), NaCl, and protein content). There was no significant difference ($p>0.05$) in physicochemical values between the predictive model and the reference method, except for the pH value. Finally, the NIR technique outperforms conventional methods in determining multiple quality parameters simultaneously (TSS, color, protein, and NaCl) for nam pla-ra with less than three minutes per scan.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

SUPPLEMENTARY MATERIALS

Supplementary materials are available at: www.ftb.com.hr.

AUTHORS' CONTRIBUTION

S. Jungtheerapanich contributed to the main conception, acquired funding, conducted formal data analysis, performed methodology and investigation, prepared the manuscript, and managed the project. S. Kasemsumran provided conceptualization, NIR methodology, guidance, and reviewing and editing of the original draft manuscript. K. Ngowsuwan assisted in NIR methodology. All authors have read and agreed to the published version of the manuscript.

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Table 1. Composition variation and properties of nam pla-ra samples used to develop PLS models

Parameter	Calibration set (*N*=75)				Prediction set (*N*=25)			
	Min	Max	Mean	S.D.	Min	Max	Mean	S.D.
w(TSS)/%	13.93	48.50	28.35	4.80	22.77	41.10	28.76	4.45
pH	4.08	6.06	5.03	0.39	4.42	5.90	5.06	0.39
*L**	25.97	36.51	32.13	2.28	28.16	36.48	32.36	2.21
*a**	1.31	7.59	3.47	0.79	2.41	6.20	3.52	0.73
*b**	1.69	10.82	6.88	1.77	3.99	9.83	7.03	1.65
w(NaCl)/%	8.38	21.87	15.80	2.62	11.35	21.57	16.08	2.49
w(protein)/%	1.57	7.82	3.50	1.13	2.06	6.94	3.59	1.16

Table 2. Band assignments of significant NIR regions of nam pla-ra

Wavelength/nm	Band assignment	Substance
1403	O-H first overtone	Water (*19*)
1463	O-H first overtone	Water (*19*)
1710–1750	first overtones of the C-H stretching	Lactic acid, Lactate (*20–24*)
1877–1900	O-H stretching, bending combination band	Water (*19*)
1890	O-H stretch combined with C=O stretch	Carboxylic acid (*24*)
2050–2168	N-H stretching combination, N-H band second overtone	Protein (*24*)
2160	C-H stretch, C=O stretch combination	Carboxylic acid (*24*)
2242	C-H stretching, CH_2 deformation combination	Starch, Sugar (*24*)
2470	C-H stretch combined with C-O stretch	Carboxylic acid (*24*)
2472	C-H stretching, C-C stretching combination, C-O-C stretching combination	Starch, Sugar (*24*)

Table 3. PLS model statistics for nam pla-ra physicochemical values

Parameter	Pretreatment	Factor	Calibration (*N*=75)			Prediction (*N*=25)		
			R^2	SEC	Bias	R^2	SEP	Bias
Full spectrum range (1000–4000 nm)								
w(TSS)/%	None	5	0.9716	0.8093	$1.767\cdot10^{-6}$	0.9756	0.6762	-0.1574
	2D (11,11,2)	5	0.9839	0.6086	$9.664\cdot10^{7}$	0.9748	0.7056	-0.0163
	SNV	4	0.9732	0.7856	$9.664\cdot10^{7}$	0.9806	0.6062	-0.1276
pH	None	22	0.9852	0.0470	$-9.549\cdot10^{-6}$	0.8096	0.1496	-0.0762
	2D (11,11,2)	13	0.9430	0.0922	$-2.352\cdot10^{-7}$	0.7677	0.1705	-0.0733
	SNV	19	0.9755	0.0605	$-3.236\cdot10^{-6}$	0.7866	0.1654	-0.0655
*L**	None	8	0.8913	0.7518	$4.578\cdot10^{-7}$	0.8146	0.9434	-0.1265
	2D (7,7,2)	4	0.7581	1.1215	$5.086\cdot10^{-7}$	0.7844	1.0266	-0.0240
	SNV	7	0.8845	0.7751	$2.797\cdot10^{-7}$	0.7951	1.0000	0.0445
*a**	None	9	0.8336	0.3239	$8.074\cdot10^{-7}$	0.7270	0.3725	0.0699
	2D (3,3,2)	4	0.7236	0.4175	$6.358\cdot10^{-9}$	0.3423	0.5885	0.0078
	SNV	13	0.9294	0.2110	$4.768\cdot10^{-7}$	0.6200	0.4457	-0.0386
*b**	None	9	0.8672	0.6435	$-1.367\cdot10^{-6}$	0.7554	0.8138	-0.0754
	2D (11,11,2)	8	0.8135	0.7626	$-1.717\cdot10^{-7}$	0.7581	0.7739	-0.2439
	SNV	6	0.8268	0.7349	$9.219\cdot10^{-8}$	0.7570	0.8147	0.0005
w(NaCl)/%	None	6	0.9822	0.3494	$-1.030\cdot10^{-6}$	0.9722	0.4116	0.0491
	2D (11,11,2)	5	0.9668	0.4763	$3.815\cdot10^{-8}$	0.7793	1.1448	-0.2284
	SNV	3	0.9728	0.4315	$-6.485\cdot10^{-7}$	0.9685	0.4410	-0.0111
w(protein)/%	None	7	0.8996	0.3576	$-2.480\cdot10^{-7}$	0.8544	0.4414	0.0091
	2D (11,11,2)	12	0.9623	0.2190	$4.292\cdot10^{-8}$	0.8292	0.4781	0.0065
	SNV	5	0.8903	0.3737	$-2.750\cdot10^{-7}$	0.8570	0.4368	0.0233

Parameter	Pretreatment	Factor	Calibration (*N*=75)			Prediction (*N*=25)		
			R^2	SEC	Bias	R^2	SEP	Bias
Selected spectrum spectrum range (1000–1889.64 and 2030.87–2408.48 nm)								
w(TSS)/%	None	6	0.9885	0.5151	$1.284\cdot10^{-6}$	0.9836	0.5677	0.0339
	2D (11,11,2)	4	0.9840	0.6069	$6.358\cdot10^{-7}$	0.9649	0.8267	0.0975
	SNV	4	0.9784	0.7064	$8.901\cdot10^{-7}$	0.9682	0.7901	0.0697
pH	None	13	0.8965	0.1243	$3.815\cdot10^{-7}$	0.6240	0.2337	-0.0378
	2D (11,11,2)	12	0.9421	0.0930	$-9.537\cdot10^{-8}$	0.8231	0.1511	-0.0584
	SNV	14	0.9084	0.1169	$-6.104\cdot10^{-7}$	0.6697	0.2191	-0.0350
*L**	None	4	0.7526	1.1343	$1.780\cdot10^{-7}$	0.6998	1.2075	-0.0993
	2D (7,7,2)	7	0.8462	0.8942	$2.391\cdot10^{-6}$	0.7949	0.9956	0.1076
	SNV	13	0.9498	0.5110	$-3.942\cdot10^{-6}$	0.8003	0.9846	-0.0829
*a**	None	12	0.9042	0.2458	$1.295\cdot10^{-6}$	0.6166	0.4481	0.0340
	2D (9,9,2)	7	0.7880	0.3656	$-1.653\cdot10^{-7}$	0.4320	0.5444	-0.0522
	SNV	11	0.9002	0.2509	$4.689\cdot10^{-7}$	0.6462	0.4316	-0.0113
*b**	None	8	0.8322	0.7234	$-6.994\cdot10^{-7}$	0.7666	0.7367	-0.3018
	2D (9,9,2)	7	0.8246	0.7395	$-5.563\cdot10^{-7}$	0.6949	0.8875	-0.2096
	SNV	8	0.8385	0.7097	$-6.358\cdot10^{-8}$	0.7117	0.8852	-0.0609
w(NaCl)/%	None	5	0.9845	0.3252	$-5.468\cdot10^{-7}$	0.9761	0.3843	0.0009
	2D (9,9,2)	5	0.9796	0.3730	$-2.925\cdot10^{-7}$	0.9164	0.7186	-0.0308
	SNV	4	0.9904	0.2566	$-2.797\cdot10^{-7}$	0.9718	0.4175	-0.0151
w(protein)/%	None	9	0.9342	0.2893	$-5.817\cdot10^{-7}$	0.8109	0.4961	-0.0812
	2D (11,11,2)	7	0.9336	0.2907	$-1.208\cdot10^{-7}$	0.8556	0.4338	-0.0695
	SNV	7	0.9256	0.3078	$8.424\cdot10^{-8}$	0.8347	0.4676	-0.0500

The best model for each parameter is shown in gray. Spectral regions for model development (selected spectrum) were 1000–1889.64 and 2030.87–2408.48 nm. Pretreatments: 2D=second derivatives, SNV=standard normal variate. PLS model statistics: R^2=coefficient of determination, SEC=standard error of calibration, SEP=standard error of prediction

Table 4. Statistics of predictive model validation for nam pla-ra physicochemical values

Parameter	Number of samples	Selected model	Bias	Sig. (2-tailed)
w(TSS)/%	25	Selected spectrum (non-pretreatment)	0.0339	0.768*
pH	25	Full spectrum (non-pretreatment)	-0.0762	0.018
*L**	25	Full spectrum (non-pretreatment)	-0.1265	0.509*
*a**	25	Full spectrum (non-pretreatment)	0.0699	0.358*
*b**	25	Selected spectrum (non- pretreatment)	-0.3018	0.052*
w(NaCl)/%	25	Selected spectrum (non- pretreatment)	0.0009	0.990*
w(protein)/%	25	Selected spectrum (2D (11,11,2) pretreatment)	-0.0695	0.432*

*Indicates no significant difference between the quality value from the reference method and the predictive model at α=0.05

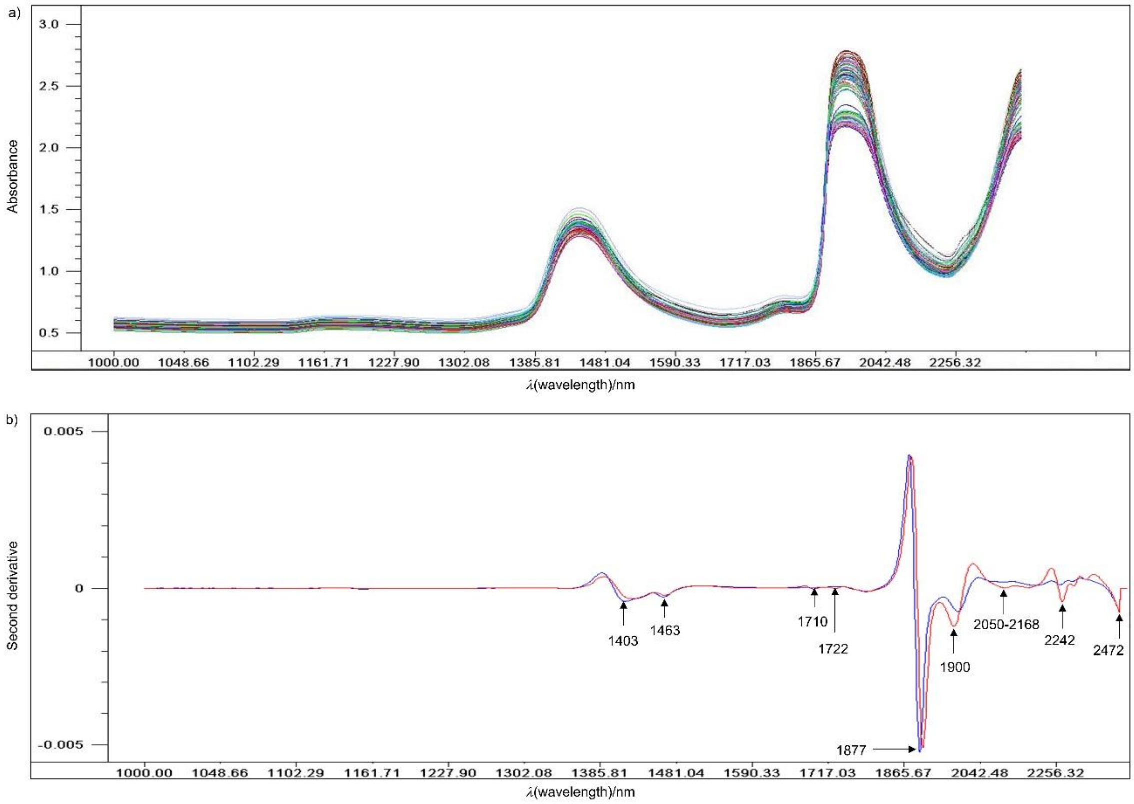


Fig. 1. a) Original NIR spectra of nam pla-ra product and b) spectra after pretreatment with second derivative Savitzky-Golay method

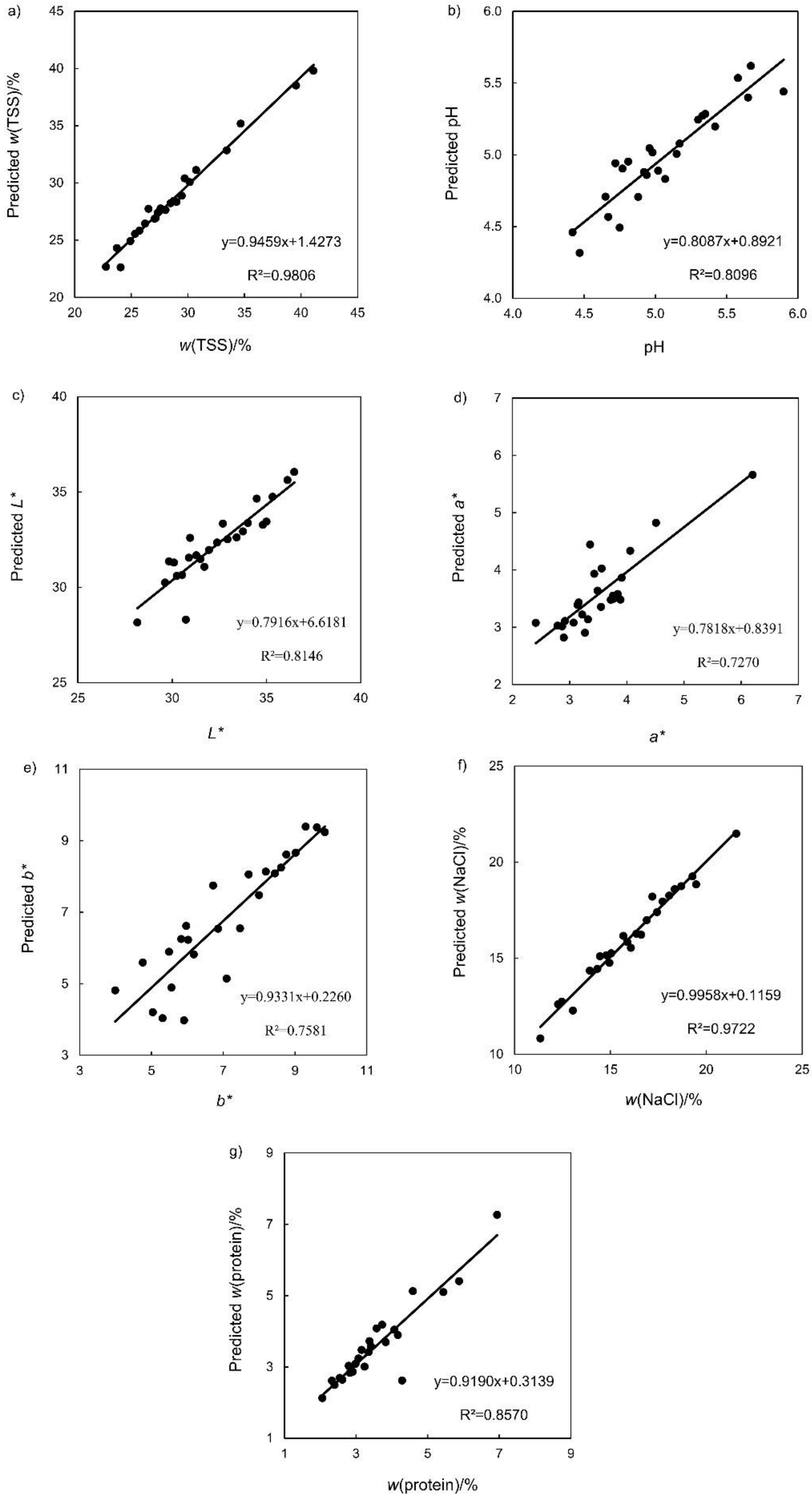
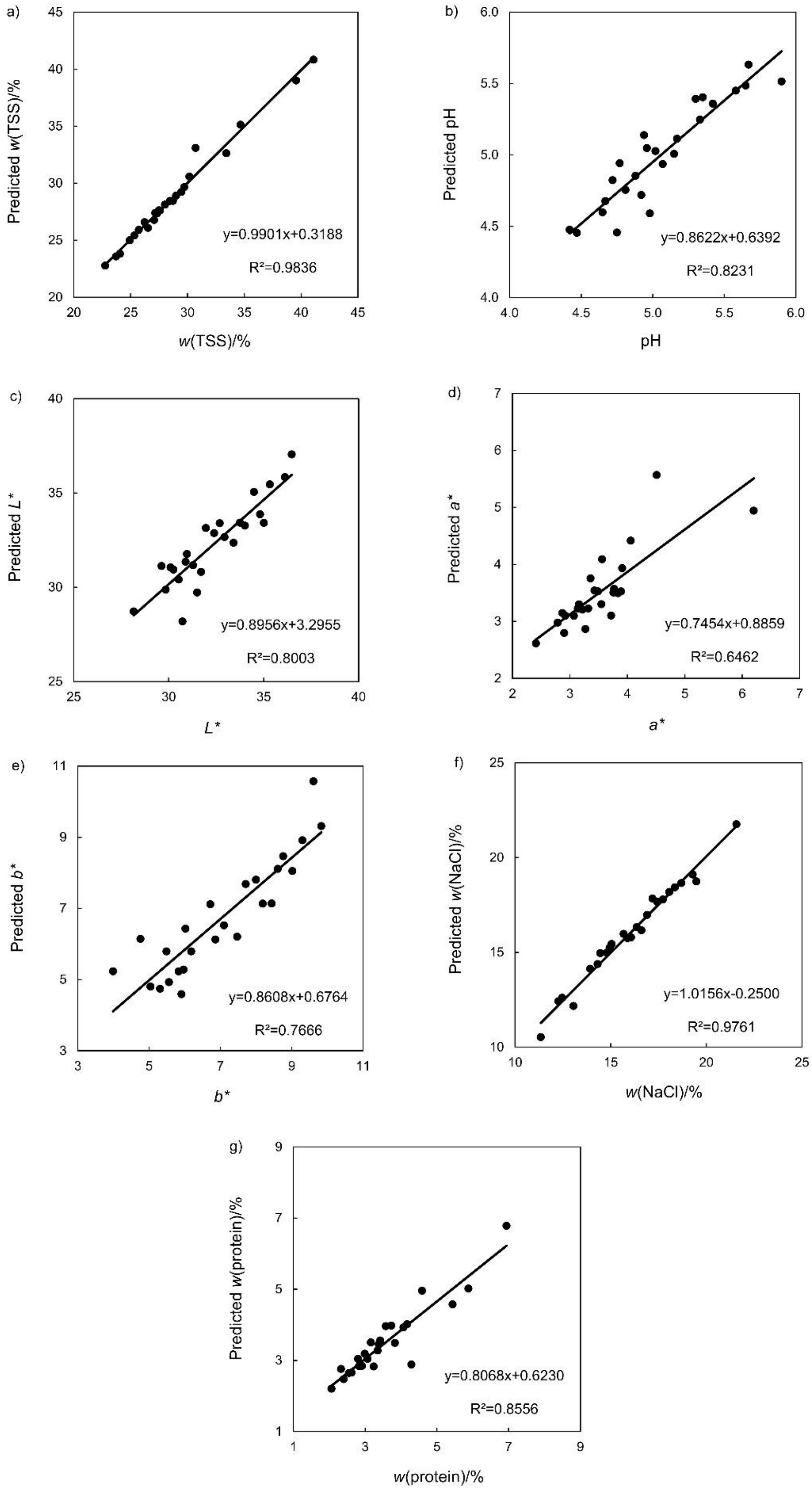

a)
Predicted w(TSS)/%
w(TSS)/%
y=0.9459x+1.4273
R²=0.9806
b)
Predicted pH
pH
y=0.8087x+0.8921
R²=0.8096
c)
Predicted L*
L*
y=0.7916x+6.6181
R²=0.8146
d)
Predicted a*
a*
y=0.7818x+0.8391
R²=0.7270
e)
Predicted b*
b*
y=0.9331x+0.2260
R²=0.7581
f)
Predicted w(NaCl)/%
w(NaCl)/%
y=0.9958x+0.1159
R²=0.9722
g)
Predicted w(protein)/%
w(protein)/%
y=0.9190x+0.3139
R²=0.8570

Fig. 2. Scatter plots for comparison of measured and predicted values (from full spectrum) for nam pla-ra qualities of prediction set for a) TSS, b) pH, c) *L**, d) *a**, e) *b**, f) sodium chloride content, and g) protein content



a)
Predicted w(TSS)/%
w(TSS)/%
y=0.9901x+0.3188
R²=0.9836
b)
Predicted pH
pH
y=0.8622x+0.6392
R²=0.8231
c)
Predicted L*
L*
y=0.8956x+3.2955
R²=0.8003
d)
Predicted a*
a*
y=0.7454x+0.8859
R²=0.6462
e)
Predicted b*
b*
y=0.8608x+0.6764
R²=0.7666
f)
Predicted w(NaCl)/%
w(NaCl)/%
y=1.0156x-0.2500
R²=0.9761
g)
Predicted w(protein)/%
w(protein)/%
y=0.8068x+0.6230
R²=0.8556

Fig. 3. Scatter plots for comparison of measured and predicted values (from selected spectrum) for nam pla-ra qualities of prediction set for a) TSS, b) pH, c) *L**, d) *a**, e) *b**, f) sodium chloride content, and g) protein content

SUPPLEMENTARY MATERIAL

Table S1. Information about nam pla-ra (manufacturer, city, province, and country of origin)

Manufacturer	Origin (city, province, country)
Zen and Kosum Interfoods Co., Ltd.	Kosum Phisai, Maha Sarakham, Thailand
Piromporn Agriculture Co., Ltd.	Lat Phrao, Bangkok, Thailand
Setthisaeb Limited Partnership	Thawat Buri, Roi Et, Thailand
Thai Kori Nomimono Co., Ltd.	Mueang Sakon Nakhon, Sakon Nakhon, Thailand
Pickled Fish Factory Mother Prakas Co., Ltd.	Sam Sung, Khon Kaen, Thailand
Mekhala Inter Foods Co., Ltd.	Mueang Samut Prakan, Samut Prakan, Thailand
Jawirat Food Co., Ltd	Phayuha Khiri, Nakhon Sawan, Thailand
Native Food Co., Ltd	Wihan Daeng, Saraburi, Thailand
Plara Racha Co., Ltd	Lat Lum Kaeo, Pathum Thani, Thailand
Phetdam Foods Co., Ltd.	Mueang Kalasin, Kalasin, Thailand
Nongporn Food Industries Co., Ltd.	Bang Bua Thong, Nonthaburi, Thailand
Plara Maerien Co., Ltd.	Erawan, Loei, Thailand
Sudjai Jarernkankha Co., Ltd.	Khanu Woralaksaburi, Kamphaeng Phet, Thailand
Yan Wal Yun Co., Ltd.	Mueang Samut Sakhon, Samut Sakhon, Thailand
Siriporn (1622) Co., Ltd	Thawat Buri, Roi Et, Thailand
K.S.F Foods Products Co., Ltd	Kosum Phisai, Maha Sarakham, Thailand
Elah Kumpang Interfoods Co., Ltd.	Kantharawichai, Maha Sarakham, Thailand
Dee Khuk Co., Ltd.	Mueang Nakhon Ratchasima, Nakhon Ratchasima, Thailand
Nit Foods Limited Partnership	Sawankhalok, Sukhothai, Thailand
Plara Chef Praitoon Food Products Co., Ltd.	Ubolratana, Khon Kaen, Thailand
Thepthida Food Co., Ltd	Prachaksinlapakhom, Udon Thani, Thailand
1999 Khon Kaen Food Products	Mueang Khon Kaen, Khon Kaen, Thailand

Table S2. Details about the mixture design technique

Mixture design technique	Sample	Proportion of sample		
		A	B	C
Simplex lattice design	1	1	0	0
	2	0	0	1
	3	0	1	0
	4	0.67	0	0.33
	5	0.67	0.33	0
	6	0.33	0	0.67
	7	0.33	0.67	0
	8	0	0.33	0.67
	9	0	0.67	0.33
	10	0.33	0.33	0.33
	11	1	0	0
	12	0	0	1
	13	0	1	0
	14	0.67	0	0.33
	15	0.67	0.33	0
	16	0.33	0	0.67
	17	0.33	0.67	0
	18	0	0.33	0.67
	19	0	0.67	0.33
	20	0.33	0.33	0.33
	21	1	0	0
	22	0	0	1
	23	0	1	0
	24	0.67	0	0.33
	25	0.67	0.33	0
	26	0.33	0	0.67
	27	0.33	0.67	0
	28	0	0.33	0.67
	29	0	0.67	0.33
	30	0.33	0.33	0.33
Simplex axial design	31	1	0	0
	32	0	0	1
	33	0	1	0
	34	0.5	0.5	0
	35	0.5	0	0.5
	36	0	0.5	0.5
	37	0.67	0.16	0.16
	38	0.16	0.16	0.67
	39	0.16	0.67	0.16
	40	0.33	0.33	0.33
	41	1	0	0
	42	0	0	1
	43	0	1	0
	44	0.5	0.5	0
	45	0.5	0	0.5
	46	0	0.5	0.5
	47	0.67	0.16	0.16

Mixture design technique	Sample	Proportion of sample		
		A	B	C
	48	0.16	0.16	0.67
	49	0.16	0.67	0.16
	50	0.33	0.33	0.33
	51	1	0	0
	52	0	0	1
	53	0	1	0
	54	0.5	0.5	0
	55	0.5	0	0.5
	56	0	0.5	0.5
	57	0.67	0.16	0.16
	58	0.16	0.16	0.67
	59	0.16	0.67	0.16
	60	0.33	0.33	0.33